

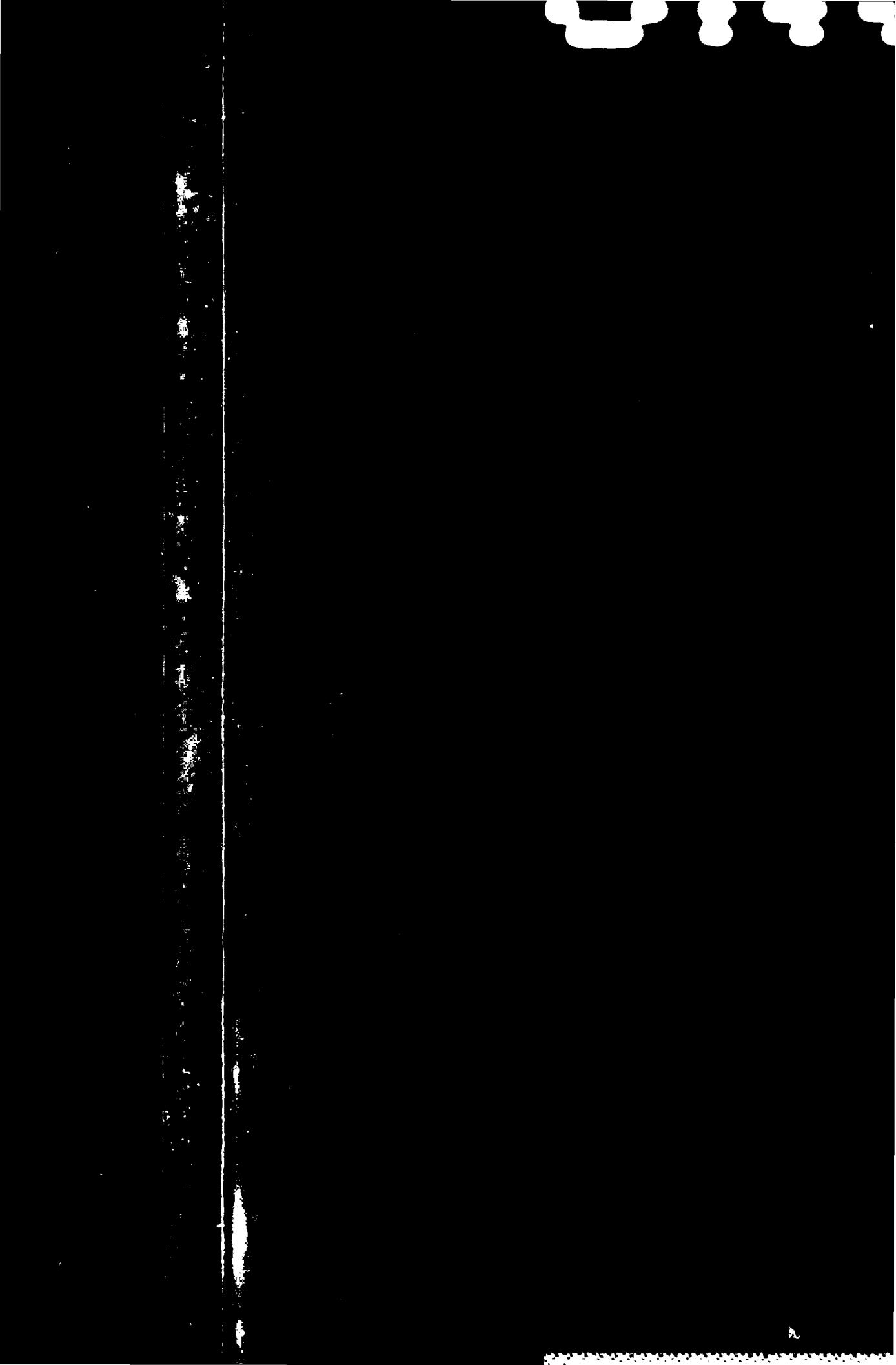
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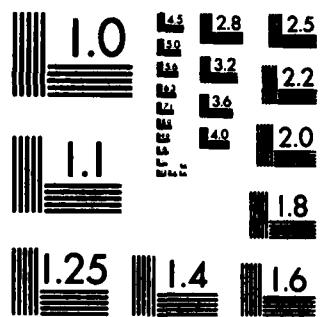
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## BATTERY MATERIALS SYMPOSIUM BRUSSELS 1983

BY LARRY E. DEVRIES

RESEARCH AND TECHNOLOGY DEPARTMENT

OCTOBER 1983

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## FOREWORD

The International (I) Common (C) Sample Office organized by the Cleveland, Ohio section of the Electrochemistry Society sponsors an international symposium on manganese dioxide every five years. The I.C. Sample Office is aware of the need to advance the science and engineering of the whole range of dry battery materials. With this goal in mind, the Battery Material Research Association (BMRA) was formed in 1983. The BMRA sponsored its first international symposium in September. The many favorable comments received after the meeting indicate that the BMRA is achieving its goal, and that the selection of papers presented was excellent.

Approved by:


  
 JACK R. DIXON, Head  
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CHAPTER 1

INTRODUCTION

A Battery Material Symposium was held in Brussels, Belgium on 14-16 September 1983. It was the first international meeting of the Battery Material Research Association. The purpose of the meeting was threefold:

- a. To discuss current research on battery materials
- b. To involve new people through discussions held in response to the papers presented
- c. To prepare for the 1985 MnO<sub>2</sub> Symposium in Graz, Austria.

Twenty countries were represented at the symposium. A wide range of topics was covered including the production and performance of battery materials, thermodynamics of electrochemical reactions, corrosion of battery materials, and rechargeable systems.

The papers dealt mainly with primary (nonrechargeable) dry cell materials. A battery cell consists of an anode, cathode, and electrolyte as the primary components with structural materials, insulators, current collectors, separators, and seals as secondary components. Figure 1 shows the structure of a typical cell. Current collectors are the electrical conductors in the anode and cathode. The zinc anode can serve as its own current collector. In Figure 1 it makes electrical contact with the metal base which is the anode (-) terminal. A carbon rod serves as the cathode current collector making electrical contact with the metal cover which is the cathode (+) terminal at the top of the cell. A separator keeps the anode from making contact with the cathode. For the cell in Figure 1 it is a gelled (paste) electrolyte. A paper disc acts as the insulator between the anode and cathode at the bottom of the cell. A plastic washer serves as the insulator at the top of the cell preventing a short circuit between the anode and cathode. The pitch seal at the top of the cell is vented to allow hydrogen from the corrosion of the anode to escape. The paper washer at the top of the cell prevents the pitch from coming into contact with the cathode. The wax seal at the top of the cell helps keep the electrolyte from drying out as hydrogen vents from the cell. The paper jacket is crimped into the metal base and into the metal cover (Figure 1) binding the cell together.

During discharge the anode is oxidized releasing electrons which flow through the current collector to the anode terminal where they enter the external circuit. Electrons leave the external circuit into the cathode terminal and current collector where they reduce the cathode. The internal circuit is completed through the electrolyte which furnishes ionic conductivity between the anode and cathode. A dry cell cannot discharge if

it is dry. The liquid (in this case aqueous) electrolyte solution is contained in the separator, porous cathode, and porous anode (when there is one) so that there is no free-flowing electrolyte in the cell.

CHAPTER 2  
CORROSION

In many battery systems, the chemically active anode materials such as zinc corrode. The corrosion is usually caused by reaction of the anode with an electrolyte component or components. Impurities in battery materials can increase or decrease the corrosion rate. Some of the materials which increase the corrosion rate of zinc are antimony, arsenic, iron, and molybdenum. Different materials cause increased corrosion rates at different concentrations (often in the low ppm or subppm range). Unfortunately, the presence of two or more elements can reduce the level at which one or more of them will cause an increased corrosion rate. The corrosion reaction is (1)



for zinc in alkaline electrolytes. Measurement of the hydrogen evolved is a direct measurement of the corrosion rate (Ref. 1,2).

One way to reduce the corrosion rate is to use chemically prepared materials instead of natural materials (which are of lower purity). This is especially true for the carbon and manganese dioxide used in batteries. Another way to reduce corrosion is to use alloys. Lead decreases the corrosion rate of zinc when alloyed in the concentration range of 500 ppm and higher. It is often used in the zinc anodes used in batteries. Another alloy of zinc containing indium as well as lead is now being studied (Ref. 2).

The most common way of reducing zinc corrosion is by forming a mercury amalgam with it. It is desirable to reduce the amount of mercury in batteries since on disposing of discharged batteries mercury can be released into the environment. However, a low level of corrosion is usually found when the mercury concentration in the zinc approaches 10%. Data were presented that indicated that a thorough amalgamation of the zinc to the center of the particles would reduce the percentage of mercury necessary to keep the corrosion rate low in alkaline electrolytes (Ref. 3). A zinc-lead-indium alloy with 1.5% mercury is claimed to keep the corrosion rate at the same level as a zinc-lead alloy with 10% mercury (Ref. 2).

## CHAPTER 3

## MANGANESE DIOXIDE

## TYPES OF BATTERIES

Manganese dioxide is used as the cathode in several types of batteries. The largest number of battery cells made in the world (several billion each year) contain a zinc anode and an ammonium chloride-zinc chloride electrolyte. This is the "acid" system known as the Leclanché battery. The alkaline version contains either sodium hydroxide or potassium hydroxide for the electrolyte. Manganese dioxide is used in magnesium batteries with either a magnesium bromide or magnesium perchlorate electrolyte. In some mercuric oxide-zinc cells manganese dioxide is added to the mercuric oxide where it discharges at a high voltage before the mercuric oxide begins to discharge. The resulting porous cathode structure acts as a "sponge" to contain the mercury formed during discharge and thus prevents mercury short circuits. The newest type of battery using a manganese dioxide cathode contains a lithium anode and nonaqueous electrolyte.

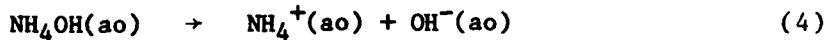
## ELECTROCHEMISTRY

The Leclanché battery has been in use for over a century. However, the chemistry and electrochemistry of the manganese dioxide cathode is still a much debated topic (Ref. 4) even though they have been studied since the early use of the battery. Manganese dioxide is a nonstoichiometric oxide with the composition  $MnO(2-x)$  where  $x$  is  $> 0$ . There are different crystalline forms of manganese dioxide which behave differently electrochemically. Their discharge performance depends on the physical and chemical treatment prior to use in cells as well as the electrolyte composition. The different closed circuit voltages under a given set of discharge conditions for different forms of manganese dioxide are a physical chemical phenomenon as are the associated variations in the discharge capacity.

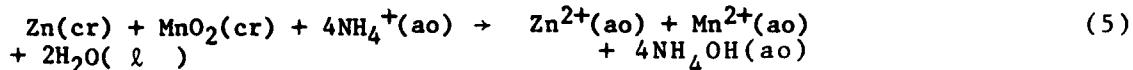
While debates are still going on about the electrochemistry of the discharge of a Leclanché cell, the overwhelming evidence is that in fresh cells with a low pH and low concentration of manganese(II) ions a two-electron reduction of manganese dioxide occurs, (2).



As I pointed out over 15 years ago and reintroduced at the Brussels symposium, the calculated open circuit voltage (OCV), 1.603 V for (2), and the measured OCV, 1.598 V, agree with each other to within 0.3% (Ref. 5). Several equilibria are involved in the system such as (3) and (4)

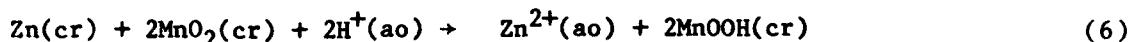


and can be used to generate thermodynamic data. Using this data voltages for other forms of the discharge reaction such as (5)



have been calculated, 1.601 V. The agreement with the voltage for (2) is within 0.1%. The two calculated values should be identical, and the difference is considered to be insignificant.

The slope of the voltage versus pH curve indicates whether a two or a one electron reduction occurs. While not a major point of two papers (Refs. 4,6) the data showed that the slope of the curve corresponded to a two-electron reduction. As discharge progresses and the pH and manganese(II) ion concentration increase, it appears that a one-electron reduction of manganese dioxide occurs according to (6)



where manganese oxide hydroxide is produced. Two papers gave detailed information on the slope of the curve. Under the initial cell conditions (low pH and low manganese(II) ion concentration) the slope corresponds to a two-electron reduction and does shift to a slope corresponding to a one-electron reduction under conditions of a progressing discharge (Refs. 7,8).

#### CHEMICALLY PREPARED MANGANESE DIOXIDE

Chemically prepared manganese dioxide is the main source of battery cathode material. It is prepared after treatment of a manganese dioxide containing ore by either a direct chemical precipitation (the CMD form) or electrolytic deposition (the EMD form). There is a difference in the electrochemical performance between the CMD and the EMD. This is believed to result from larger pores in the CMD particles than in the EMD particles. Concentration polarization should be less severe with larger pores (Ref. 9). Thus not only is the porosity of the cathode mix important, but the porosity of the particles in the mix is also important.

The EMD is the major form of chemically prepared manganese dioxide used in batteries. Its performance depends on the treatment given it prior to use. Studies on the heat treatment of the EMD indicate that between 478 and 723 K there is a loss of lattice water. The discharge performance falls off sharply when the EMD is heated to between 673 and 723 K (Refs. 4,8). Under low pH conditions with low concentrations of manganese(II) ions, the slope of the voltage versus pH curve corresponds to a two-electron reduction of manganese dioxide and is independent of the temperature treatment. At higher pH's where a one-electron reduction can occur, the slope of the curve is affected by the temperature treatment (Ref. 8).

The first step in one method of production of EMD is the thermochemical or electrochemical reduction of a manganese dioxide ore to the +2 oxidation state of manganese. A solution of manganese(II) ions is purified and the manganese(II) ions are oxidized forming a deposit of manganese dioxide on the anode. Carbon and lead have been used for anodes. However, carbon electrodes do not have long-term mechanical and chemical stability; lead electrodes, while having longer useful lives, contaminate the manganese dioxide with lead. Titanium electrodes have long-term mechanical and chemical stability. To have a high rate of deposition of manganese dioxide, a high current density is needed. A porous titanium electrode has been developed to maintain a high current density,  $130 \text{ A/m}^2$  (Ref. 10).

A second method of producing EMD is a one-step, electrolytic process. The manganese dioxide is used as a slurry. It is reduced to manganese(II) ions at a carbon cathode and oxidized and deposited as manganese dioxide on a titanium anode. Iron is an impurity in the ore used, and it is believed to aid in maintaining sufficiently high current densities by iron(II) reducing part of the manganese dioxide. The iron(III) ions formed are reduced back to iron(II) ions at the cathode. The overall slurry electrolysis is limited by the diffusion of iron(III) ions to the cathode. With a stirred catholyte, a  $100 \text{ A/m}^2$  current density is obtained (Ref. 11). However, because it is a one-step process, the manganese dioxide purity will not be as high as for the multistep process with a purification step.

CHAPTER 4

ELECTROLYTE

In the Leclanché battery, the electrolyte is normally an aqueous solution of zinc chloride and ammonium chloride. However, an attempt is being made to increase the use of an aqueous solution containing only zinc chloride for the electrolyte. The stated reason is that the zinc chloride gives better high current discharges (Ref. 12). The electrolyte enhances corrosion, and a problem has been to prevent electrolyte leakage from sealed cells. Even the "high density" carbon rod current collector has to be made hydrophobic so that the electrolyte will not penetrate it and cause corrosion of the cathode plate at the top of the cell.

CHAPTER 5

SEPARATORS

The primary purpose of separators is to keep the anode and cathode from coming into contact which would result in their direct chemical rather than electrochemical reaction. The separator can be made from a wide variety of materials including Kraft paper, cellophane, and polypropylene. The structure may be woven, nonwoven, pulp, or felt among others. The surface of the material may be treated with a starch paste or other material. Gelled (paste) electrolytes are also used as separators. In addition to providing separation, separators are also used to absorb and contain electrolyte, permit ionic conduction between the anode and cathode, and prevent dendrite growth into the cathode. Often a single type and thickness of separator will not accomplish all of the functions necessary in a particular cell so as many as five layers of different materials may be used. The nongelled electrolyte separators need strength (the ability to be wrapped or folded without breaking or tearing), abrasion resistance, and chemical inertness to the internal cell components. Several types of treated and nontreated paper separators were described (Ref. 13) as well as separators made from a new separator material, polyvinyl alcohol (Ref. 14).

## CHAPTER 6

## CARBON FOR MANGANESE DIOXIDE CATHODE

The cathode mix usually contains the active material (manganese electrolyte to furnish ionic conductivity, and carbon to give it electrical conductivity. Synthetic carbon has mainly replaced natural carbon. It permits higher purity (lower metal contamination) carbon to be used eliminating the chief problem with natural graphite. The two forms of synthetic carbon used are carbon black and graphite. The carbon black is used in cells with the ammonium chloride-zinc chloride or zinc chloride electrolyte. The synthetic graphite is used in cells containing an electrolyte.

One of the carbon blacks, acetylene black (ab), has had the most extensive use in the battery industry. The prime method of manufacture is thermal decomposition of acetylene in the absence of air. High purity material is obtained by using pure acetylene gas (Ref. 15) the material having good electrical conductivity and good electrolyte retention. The structure is like a three-dimensional net or fibrous sponge. The structure can be destroyed resulting in the loss of electrolyte retention if care is not taken in forming the cathode mix.

Another source of carbon black under development as an alternative to ab is oil. The materials are formed as a by-product of a partial process used to produce synthesis in gas and ammonia. The material has a chain structure and is sensitive to handling during the cathode mix preparation as is ab. Metal impurities in the oil feed stock such as nickel, and vanadium end up in the carbon (typically 80 ppm for iron for nickel, and 80 ppm for vanadium). For both types of carbon black, discharge performance of a cell depends on the electrolyte composition and amount of carbon in the cathode. On the average, cells with ab have slightly better discharge performance than those with oil carbon black. Tests such as short circuit current, 4  $\Omega$  continuous discharge, and 5  $\Omega$  -2hr/day discharge (Ref. 16). Both forms of carbon black cause hydrogen to be produced by zinc corrosion with the ab forming slightly more hydrogen. Both forms of carbon black also produce carbon dioxide from oxidation of the carbon in the cathode mix with ab forming more CO<sub>2</sub> for a short time after the cell is formed (at 20 hr, 21.2 mL versus 18 mL and at 60 hr, 15 mL versus 12 mL).

Synthetic graphite can be produced from either primary (virgin) or secondary (by-product) raw materials. The primary material is stated to be better for battery applications than the secondary material. The primary synthetic graphite is produced by heating virgin raw material to 300°C. As already mentioned, synthetic graphite is used in the cathode of the

manganese dioxide-zinc cell. In addition to giving electrical conductivity to the cathode mix, the synthetic graphite gives lubricity to the manganese dioxide making it more compactible and reducing tool wear from the abrasive manganese dioxide. The discharge performance of the cathode depends on the graphite's porosity, crystallinity, particle size distribution, particle shape, ash content, and chemical purity (Ref. 17). Particle size and perfection of crystallites affect electrical conductivity. In addition to enhancing corrosion of the anode, impurities also enhance oxidation of the carbon in the cathode causing it to gas ( $\text{CO}_2$ ); and this is not wanted. High purity primary synthetic graphite produces much less gas than natural graphite (at 200 hr, 1.5 mL versus 4 mL).

CHAPTER 7

RECHARGEABLE MANGANESE DIOXIDE-ZINC CELLS

The secondary (rechargeable) alkaline manganese dioxide-zinc cell has been under development for several years. Problems with both the anode and cathode have occurred after a limited number of cycles. Two papers indicate that it is now possible to obtain 100 cycles (Refs. 18,19). This is accomplished by shallow discharging of the cell to no lower than 0.9V or to a capacity of about 1/3 the nominal value. While the nickel-cadmium system can be made as a sealed unit by having the gas produced on charging recombine chemically within the cell, this has not yet been done with the manganese dioxide-zinc system (Ref. 18). One type of cathode has the manganese dioxide codeposited with the carbon. It was stated that such a cathode results in better discharge because of better bonding between the manganese dioxide, carbon, and the current collector (Ref. 19).

CHAPTER 8

CONCLUSION

From the discussions generated by the papers presented, the symposium was a success. Some new ideas were introduced dealing with old theories and problems. The information indicated where much research is needed. Gibbs energies of formation are needed on the different types of manganese dioxide and manganese oxide hydroxide. When the initial thermodynamic conditions found in fresh commercial Leclanché cells are maintained in the laboratory, twice the discharge capacity normally obtained is achieved. Work needs to be done on modifying the electrolyte so as to maintain the proper thermodynamic conditions in the cell in order to double its discharge capacity.

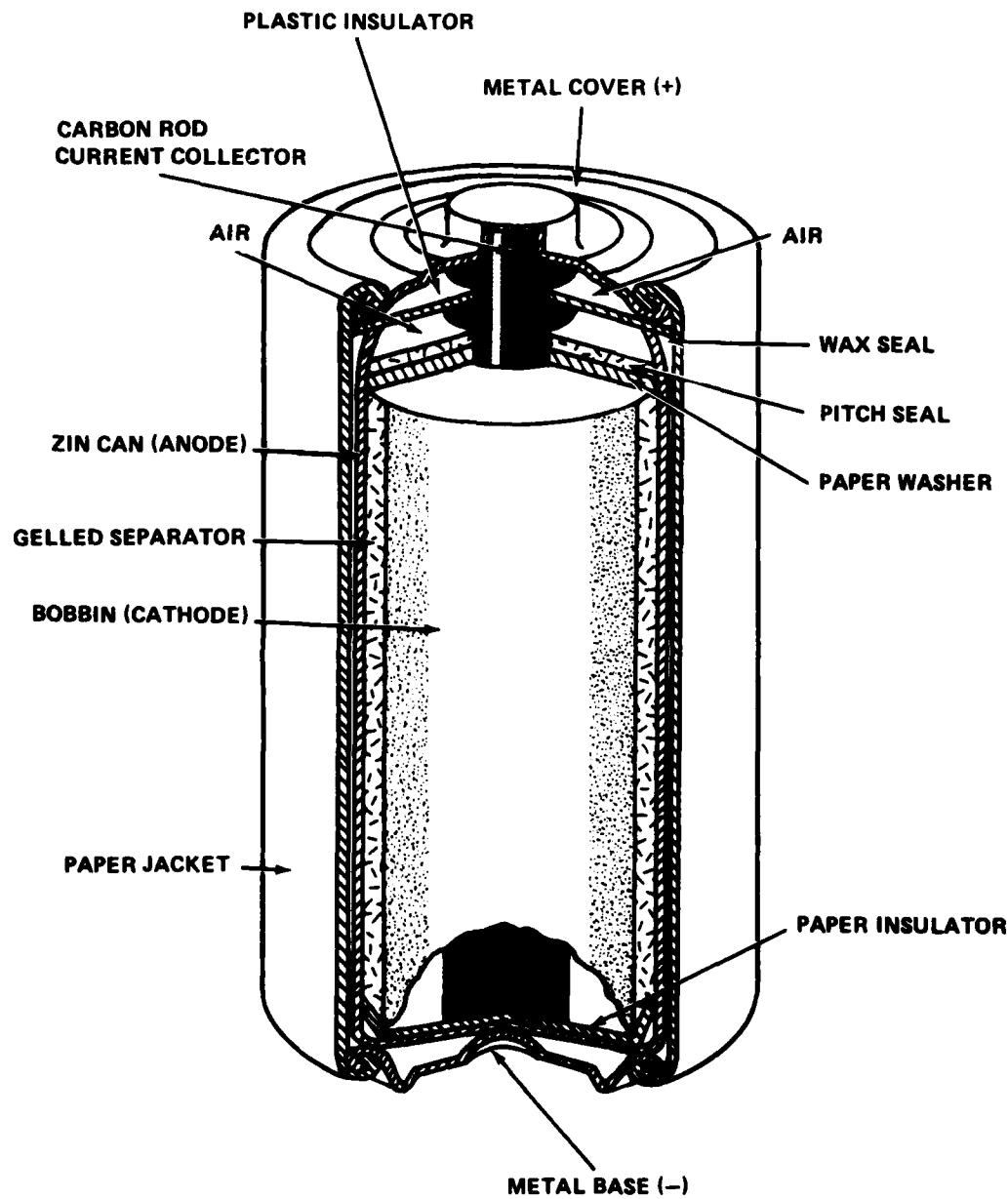


FIGURE 1. A TYPE OF DRY CELL CONSTRUCTION (LE CLANCHE' CELL).

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